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A process for the manufacturing of aluminum oxide particles, an aluminum oxide powder that is produced following said process, and the application of said powder.

The invention is concerned with a process for the manufacturing of aluminum oxide powder that is produced following said process, and the application of said powder.

The invention is concerned with a process for the production of sintering active, mainly spherical aluminum oxide particles that possess a median particle dimension of < 1.0 µm, and it is also concerned with an aluminum oxide powder that is produced following said process, as well as with the utilization of said products.

Aluminum oxide powders are utilized as pigments, with grinding and polishing medias, with fire retardant or fire resistant products, in ceramics, as a catalyst material, or as filler substances, as well as for coatings.

Determining for the technical utilization of aluminum oxide are its chemical stability, its good mechanical characteristics, specifically its advantageous wear characteristics, its high electrical resistance, and its good temperature stability. Additionally, aluminum oxide is non-toxic.

The following characteristics are required for the production of high quality ceramic, specifically for fire resistant products:

- high sintering activity (specifically due to small particle dimension)
- minimization of impurities that advance the undesirable particle growth or hinder the sintering process,
- as low as possible content of additives that create the melting phase,
- easy to process (pressing into shapes),
- high green density.

In order to achieve a high green stand resistance (green density), a limited porosity of the individual particles (powder particles) is also required.

A number of thermal, wet chemistry, and physical processes are known for the production of sintering active, microcrystalline Al_2O_3 particles and powders.

Belonging to these processes are the thermal degradation and subsequent calcination of purified alaun (ammonia aluminum sulfate), or the thermal degradation of aluminum chloride following the so-called spray screen process. The disadvantages with thermal conversion processes of this nature that are utilized with aluminum salts are the high costs for the applicable machinery, as well as the salt remnants that are present, and that can add to an increased particle growth during the sintering process.

For the production of small particle aluminum oxide, it is also known to grind up alumina that is produced following the so-called Bayer process. However, this fine grinding is very involved and takes a long time, and said time duration depends on the degree of the dimension of said small particles. This means, that particles that are sized smaller than 1 µm – for the case that they can be produced at all – can only be produced with extremely high technical involvement. Furthermore, the morphology of the such produced powder particles is splintery grainy. This can lead to disadvantages concerning the rheological characteristics.

Known from the US-A-4,818,515 is the production of spherical Al_2O_3 particles by means of a process with which water containing aluminum oxide will be subject to a multi stepped thermal treatment. Said process requires a feedstock that can only be produced with high costs involved due to its purity.

Finally, it is also possible to produce small particle calcinates by means of hydrolysis of aluminum alkoxides. However, said substances partially possess quite a high micro porosity depending on the degree of calcination. Said porosity will lead in a subsequent sintering process to a relevant (undesirable) waning.

Because of these facts, and also because of technical and economical reasons, the mentioned processes were not able to establish themselves for mass applications. Because of this, the scope of the presented invention is to provide a process that enable the production of very small aluminum oxide particles in the sub micron-range (< 1 µm) in a relatively economical manner, with which a mostly spherical particle shape, a low porosity, and thus a good ability to achieve high densities and good sintering characteristics are strived for, to enable, respectively optimize its application for the above mentioned purposes.

This scope will be achieved with the support of the process that is mentioned in the beginning, and that is characterized by means of the following steps:

- introduction of an aluminum carrier such as Al or Al_2O_3 into an oven apparatus
- heating of said aluminum carrier,
- reduction of said aluminum carrier, for the case that it was not introduced in the form of metallic aluminum, into metallic aluminum and/or aluminum carbides (including aloxi carbides),
- increasing of the oven temperature to a value, at which said metallic aluminum, respectively, the aluminum carbides will be vaporized,
- subsequent oxidation, inside of a gas flow, of the metallic aluminum, respectively, of its carbides into aluminum oxide, and
- introduction of said gas flow into a filter, while
- the temperature, atmosphere, and resting time of the aluminum oxide particles inside the gas flow will be adjusted according to the desired particle dimension.

Starting with, for example, pieces of aluminum oxide, said material thus will initially be reduced to metallic aluminum and/or aluminum carbides, which consequently will be, for example, vaporized parallel to each other, and which eventually will be re-oxidized in a suitable manner prior to the separation inside of a filter of the aluminum oxide particles secondarily created. in such a manner

Herewith, it is very important for achieving the tiny dimensions of the aluminum oxide particles that are part of the scope of the invention that are present in the gas flow will be introduced into the down stream installed filter. The shorter the lingering duration of the Al_2O_3 particles in the gas flow, the tinier the particle dimension that can also be controlled secondarily by means of the temperature and the (oxidizing) atmosphere of the gas flow.

Proven as being specifically advantageous as an oven apparatus is an electric arc oven. According to one execution example of the invention, said oven will be operated with current densities between 10 and 50 A/cm², and in the herewith specifically preferred range between 15 and 30 A/cm².

For the increase of effective vaporization performance, the addition of a reduction agent (such as carbon) showed to be advantageous with the reduction reaction of aluminum oxide to aluminum, aluminum carbide, or aluminumoxi carbide. Herewith, it is also possible to utilize carbon dispensing compounds for this purpose.

The subsequent oxidation of the steam like aluminum and/or of the condensed aluminum particles can be exhilarated by means of supplying external oxygen into the system. Herewith, it will become possible that the particles need to stay in the oxidation phase only for a short time duration, and that following said time duration, they can be precipitated immediately in a suitable filter, for example in a hose filter.

Alternatively, it is possible that the oxidation step is created in such a manner that aluminum particles are introduced into an oven section in which an oxidizing atmosphere is present.

It is possible with the support of the described process to produce sintering active, spherical aluminum oxide particles that possess a density of 3.97 g/cm ^{3} and a specific surface that ranges between 0.5 and 60 m^2/g.
The process allows for the creation of aluminum oxide particles that possess a particle size of clearly less than 1 μm, and by means of relevant adjustments of the process parameter such as temperature, atmosphere and lingering duration of the Al_2O_3 particles in the gas flow, the particle size can be reduced down to 0.10 μm.

A specific advantage consists of the fact that the Al_2O_3 particles that are produced following the described process possess an almost ideal spherical (ball shaped) configuration, which allows for the specifically advantageous utilization, for example, for grinding and polishing media, or in fire resistant ceramic materials (herewith also as a binding agent, respectively, as a component of a binding agent. The ball shape is mainly responsible for the circumstance that the particles also add to outstanding rheological characteristics of relevant systems.

For the case that an electric arc oven will be utilized, it is possible that the introduction material can be present without any problems in the shape of large pieces. Herewith, the vaporization performance of the occurring electric arc depends on its energy content and the local electric arc temperature. With current densities in the range of 10 to 50 A/cm^2, the vaporization performance is in the range of 40 to 100 g Al_2O_3/kwh.

The composition of the Al_2O_3 particles that are obtained with the support of this process depends on the utilized aluminum containing raw material (aluminum carrier), and on the utilized reduction agent. For the case that the raw material, and/or the reduction agent contain alkali, and/or earth alkali oxides, SiO_2, ferric oxide, or similar substances, the impurities can be found again almost quantitative in the final product. For the case that carbon is utilized as a reduction agent , partially also because of the carbon in the electrodes of the oven, small amounts of carbon will be released , or carbides or oxicarbides will be created. For the case that the re-oxidation does not proceed fully, small amounts of carbon, of up to about 0.5 percent by weight , can be found in the final product, which can be reduced furthermore by means of a following thermal treatment (for example, by means of a glow treatment) if so desired.

In the following, the invention is described in more detail and with the support of an execution example:

A mixture consisting of 85 weight parts of aluminum pieces, and 15 weight parts of graphite grid is introduced into an electric arc oven that is equipped with graphite electrons. Subsequent to the creation of the electric arc a melt sump is created initially that consists of aluminum oxide, Al_4O_4C, and Al_4C_3 that is of advantage as a protective layer for the floor structure of the oven (that consists herewith of carbon stones). The energy of the electric arc lays in the range between 150 and 180 kVA. The current density ranges between 16 and 23 A/cm².

Subsequently, a vaporization of the introduced material pieces occurs, while metallic aluminum and aluminum carbides are created that consequently will be re-oxidized by means of the atmosphere, or with the support of additional oxygen introduction. Following this process the material will be introduced into a fabric filter, in which the they precipitate by more than 99 percent by weight. The average size of the obtained aluminum oxide powder particles lays at 0.2 µm, and said particles possess a mostly spherical shape. The density of the material lays by 3.8 g/cm³. The specific surface area lays by 9.8 m²/g.

With the composition of the introduced Al_2O_3 material of:

0.03 percent by weight of $Na_2O + K_2O$
0.014 percent by weight of Fe_2O_3
0.03 percent by weight of MgO
0.03 percent by weight of SiO_2
and the remainder is Al_2O_3

a solid Al_2O_3 powder with the following particle composition is obtained:

0.037 percent by weight of $Na_2O + K_2O$
0.03 percent by weight of Fe_2O_3
0.05 percent by weight of MgO
0.08 percent by weight of SiO_2
0.37 percent by weight of C
and the remainder is Al_2O_3

The increase of the impurities comes from the ash contingent of the graphite that was used for the reduction, as well as from the electrodes of the oven.

Patent Claims

1. A process for the production of sintering active, mainly spherical aluminum oxide particles that possess a median particle dimension of < 1.0 µm, preferably of < 0.5 µm with the following steps:

   1.1 Introduction of an aluminum carrier, such as metallic aluminum, or aluminum oxide into an oven apparatus,

   1.2 Heating of the aluminum carrier,

1.3 Reduction of the aluminum carrier, for the case that non-metallic aluminum was introduced, into metallic aluminum, and/or aluminum carbides,

1.4 Increasing of the oven temperature to a value, at which the metallic aluminum, and/or the aluminum carbides will vaporize,

1.5 Subsequent oxidation of the metallic aluminum, respectively its carbides into aluminum oxide inside the gas flow, and

1.6 Introduction of said gas flow into a filter, with which

1.7 The temperature , atmosphere, and lingering time of the aluminum oxide particles inside of said gas flow is adjusted to correspond with the desired particle dimension.

2. A process according to claim 1 with the requirement that the aluminum carrier is introduced in the form of material pieces.

3. A process according to the claims 1 or 2 with the requirement that the vaporization occurs inside of an electric arc oven.

4. A process according to claim 3 with the requirement that the current density ranges around 10 to 50 A/cm².

5. A process according to claim 4 with the requirement that the current density ranges around 15 to 30 A/cm².

6. A process according to one of the claims 1 through 5 with the requirement that carbon, or carbon releasing compounds are utilized as reducing agent.

7. A process according to one of the claims 1 through 6 with the requirement that oxygen will blow into the gas flow during the oxidation step.

8. A process according to one of the claims 1 through 6 with the requirement that the oxidation of the vaporized aluminum, respectively, of the aluminum carbide into aluminum oxide will occur by means of introducing the aerosol into an oven section that contains an oxidizing atmosphere.

9. A process according to one of the claims 1 through 8 with the requirement that the precipitation of the aluminum oxide particles occurs inside of a hose filter.

10. Sintering active, mostly spherically shaped aluminum oxide powder that is produced according to one of the claims 1 through 9, characterized in such a way that it possesses a density between 3.2 and 3.97 g/cm³, and a specific surface of 0.5 to 60 m²/g.

11. An aluminum oxide powder according to claim 10, characterized in such a way that it possesses a specific density between 3.2 and 3.97 g/cm³, and a specific surface between 4 and 20 m²/g. b

12. An aluminum oxide powder according to claim 10 or 11 with a median particle size between 0.05 and 0.3 µm.

13. Utilization of an aluminum oxide powder according to the claims 10 through 12 as a grinding or polishing agent.

14. Utilization of an aluminum oxide powder according to the claims 10 through 12 as a binding agent in fire resistant ceramic materials.

15. Utilization of an aluminum oxide powder according to the claims 10 through 12 as filler material.
16. Utilization of an aluminum oxide powder according to the claims 10 through 12 as catalyst material.

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